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Monopotassium octapyridinium tris[hexachlorobismuthate(III)]

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Key indicators

Single-crystal X-ray study
T = 223 K
Mean σ(C–C) = 0.013 Å
R factor = 0.032
wR factor = 0.078
Data-to-parameter ratio = 25.2

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

The title compound, $K(C_5H_6N)_8[BiCl_6]_3$, contains discrete $[BiCl_6]^{3-}$ anions, and pyridinium ($C_5H_6N^+$) and potassium cations. The anion exhibits a slightly distorted octahedral geometry. K is coordinated by six Cl atoms from $[BiCl_6]^{3-}$ units. The protonated N atoms are involved in hydrogen bonding with Cl atoms, giving a three-dimensional structure.

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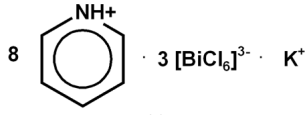
Comment

Some pyridinium halogenometallates, such as $[HPy]_2[MnX_4]$ (HPy = $[C_5H_6N^+]$; *X* = Cl, Br) (Brassy *et al.*, 1976), $[HPy]_{2.5}[FeCl_4]Cl_{1.5}$ (James *et al.*, 1982), and $[HPy]_2[ReCl_6]$ (Mrozinski *et al.*, 2002), have been investigated for their magnetic properties.



(I)

The title compound, (I), is composed of discrete K^+ and $[C_5NH_6]^+$ cations, and $[BiCl_6]^{3-}$ anions. The three independent Bi^{3+} atoms lie at the centers of $[BiCl_6]$ octahedra, with Bi—Cl distances ranging from 2.590 (2) to 2.857 (2) Å. K^+ is linked to atoms Cl26, Cl21^{i}, Cl22^{ii}, Cl31, Cl32 and Cl35, resulting in a distorted coordination arrangement, with K—Cl distances in the range 3.066 (2)–3.236 (2) Å (symmetry codes given in Table 1). Part of the crystal structure is illustrated in Fig. 1. The protonated N atoms are involved in hydrogen bonding, with N—H···Cl distances in the range 2.45 (2)–2.83 (2) Å, resulting in a three-dimensional structure (see also

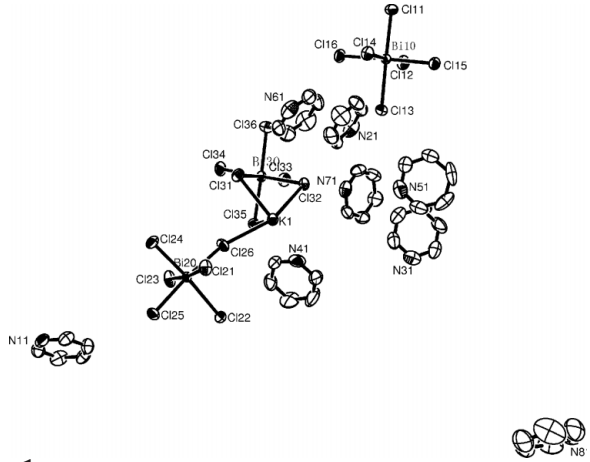


Figure 1
The asymmetric unit of (I). Displacement ellipsoids are drawn at the 50% probability level. H atoms have been omitted for clarity.

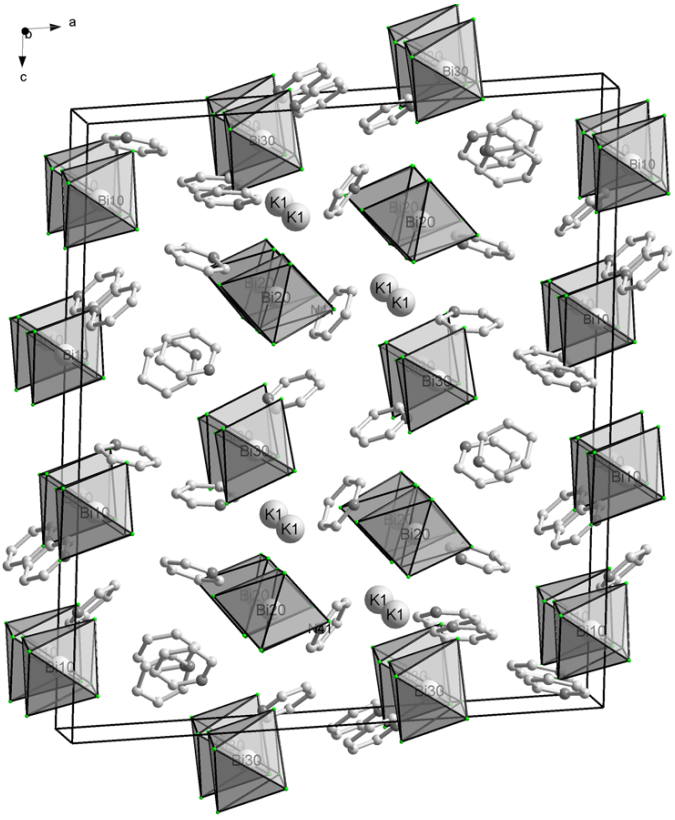


Figure 2
The crystal packing of the title compound, viewed approximately along [010]. H atoms have been omitted for clarity.

Table 2). The packing of the crystal structure is illustrated in Fig. 2.

Experimental

Pyridine (8 mmol, 0.6328 g), KCl (1 mmol, 0.0746 g) and $BiCl_3$ (3 mmol, 0.946 g) were dissolved in dilute HCl (10 ml, 6 *M*) and the resultant solution was evaporated slowly at room temperature. The title compound was obtained as prismatic colorless crystals after several days. The density of the crystals was measured by the Archimedes method.

Crystal data

$K(C_5H_6N)_6[BiCl_6]_3$
M_r = 1945.00
Monoclinic, $P2_1/c$
a = 24.392 (8) Å
b = 9.278 (3) Å
c = 28.403 (10) Å
β = 94.979 (7)°
V = 6404 (4) Å^3
Z = 4
D_x = 2.017 Mg m^{-3}
D_m = 2.016 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 15911 reflections
θ = 0.8–28.3°
μ = 9.08 mm^{-1}
T = 223 (2) K
Prism, colorless
0.15 × 0.10 × 0.09 mm

Data collection

Bruker SMART APEX CCD diffractometer
ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.350, T_{max} = 0.442
86329 measured reflections
15911 independent reflections
12299 reflections with $I > 2\sigma(I)$
R_{int} = 0.055
θ_{max} = 28.3°
$h = -32 \rightarrow 32$
$k = -12 \rightarrow 12$
$l = -37 \rightarrow 37$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.032
$wR(F^2)$ = 0.078
S = 1.04
15911 reflections
631 parameters
H atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0322P)^2 + 9.2626P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.004
$\Delta\rho_{max}$ = 1.35 e Å^{-3}
$\Delta\rho_{min}$ = −0.72 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Bi10—Cl11	2.6597 (15)	Bi30—Cl35	2.6663 (15)
Bi10—Cl14	2.6921 (15)	Bi30—Cl34	2.6674 (16)
Bi10—Cl12	2.7077 (15)	Bi30—Cl33	2.6815 (16)
Bi10—Cl15	2.7106 (15)	Bi30—Cl31	2.7152 (15)
Bi10—Cl16	2.7111 (15)	Bi30—Cl32	2.7397 (15)
Bi10—Cl13	2.7471 (15)	Bi30—Cl36	2.7507 (16)
Bi20—Cl24	2.5900 (15)	K1—Cl26	3.066 (2)
Bi20—Cl23	2.6815 (15)	K1—Cl21^{i}	3.0747 (19)
Bi20—Cl25	2.6855 (16)	K1—Cl31	3.099 (2)
Bi20—Cl21	2.7086 (15)	K1—Cl32	3.116 (2)
Bi20—Cl26	2.7238 (16)	K1—Cl22^{ii}	3.1283 (19)
Bi20—Cl22	2.8569 (15)	K1—Cl35	3.236 (2)
Cl14—Bi10—Cl12	178.03 (5)	Cl34—Bi30—Cl32	179.38 (5)
Cl11—Bi10—Cl16	90.36 (5)	Cl35—Bi30—Cl36	174.44 (5)
Cl15—Bi10—Cl16	174.64 (4)	Cl31—Bi30—Cl36	88.40 (5)
Cl11—Bi10—Cl13	176.32 (5)	Cl26—K1—Cl21^{i}	109.38 (5)
Cl23—Bi20—Cl21	172.09 (5)	Cl26—K1—Cl31	82.62 (5)
Cl25—Bi20—Cl26	172.01 (5)	Cl31—K1—Cl32	74.87 (5)
Cl21—Bi20—Cl26	88.99 (5)	Cl21^{i}—K1—Cl22^{ii}	109.71 (6)
Cl24—Bi20—Cl22	172.87 (5)	Cl31—K1—Cl35	70.82 (5)
Cl33—Bi30—Cl31	176.87 (5)	Cl32—K1—Cl35	72.41 (4)

Symmetry codes: (i) $1-x, \frac{1}{2}+y, \frac{1}{2}-z$; (ii) $1-x, y-\frac{1}{2}, \frac{1}{2}-z$.

Table 2
Hydrogen-bonding geometry (Å, °).

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N11—H11···Cl13^{iii}	0.87	2.45	3.208 (5)	146
N21—H21···Cl16^{iv}	0.87	2.64	3.333 (6)	137
N21—H21···Cl11^{v}	0.87	2.83	3.430 (6)	128
N31—H31···Cl36^{vi}	0.87	2.73	3.388 (7)	134
N41—H41···Cl26^{ii}	0.87	2.56	3.325 (6)	147
N41—H41···Cl23^{ii}	0.87	2.76	3.342 (6)	126
N51—H51···Cl23^{ii}	0.87	2.45	3.255 (6)	153
N61—H61···Cl32^{vii}	0.87	2.69	3.359 (7)	134
N61—H61···Cl21^{ii}	0.87	2.76	3.361 (7)	128
N71—H71···Cl22^{ii}	0.87	2.46	3.228 (6)	147
N81—H81···Cl36^{viii}	0.87	2.46	3.321 (8)	170

Symmetry codes: (ii) $1-x, y-\frac{1}{2}, \frac{1}{2}-z$; (iii) $1+x, y, z$; (iv) $x, 1+y, z$; (v) $-x, 1-y, -z$; (vi) $x, \frac{3}{2}-y, \frac{1}{2}+z$; (vii) $x, y-1, z$; (viii) $x, y, 1+z$.

The pyridinium H atoms were constrained to an ideal geometry, with C—H distances of 0.94 Å and N—H distances of 0.87 Å, and with U_{iso}(H) = 1.2U_{eq}(C,N). The maximum residual electron density was found 0.83 Å from atom Bi30 and the minimum electron density 1.46 Å from Bi10.

Data collection: *SMART* (Bruker, 1998); cell refinement: *SAINT* (Bruker, 1998); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 1998); program(s) used to refine structure: *SHELXTL*; molecular graphics: *ORTEP*-3 (Farrugia, 1997) and *DIAMOND* (Crystal Impact, 2000); software used to prepare material for publication: *SHELXTL*.

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